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The goal of this work was to develop and optimize an electromechanical joining technique to facilitate the creation of a periodic three dimensional open cell bulk metallic glass foam structure. As stated in the proposal, the relevant task were:
Design and construct an electromechanical joining system to join cross stacked BMG wires or wire mesh layers via homogenous flow at points of electrical contact. Characterize the microstructure and strength of the joint interface. Examine the mechanical behavior of the mesh and foam structures produced by this technique in particular the room temperature tensile and compressive strength and plastic deformation and compare these results with open cell foam models. Extend the technique to the production of other more complex lattice geometries. While the first two objectives were met, meshes and three dimensional foams were not produced due to technical difficulties and delays with the development of the joining system.

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Contract Information

Contract Number	FA9550-06-1-0213
Title of Research	Production of Open Cell Bulk Metallic Glass Foam Structures via Electromechanical Forming
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Technical Objectives

The goal of this work was to develop and optimize an electromechanical joining technique to facilitate the creation of a periodic, three dimensional open cell bulk metallic glass (BMG) foam structure. As stated in the proposal, the relevant tasks for this work were:

- Design and construct an electromechanical joining system to join cross-stacked BMG wires or wire mesh layers via homogenous flow at points of electrical contact.
- Characterize the microstructure and strength of the joint interface.
- Examine the mechanical behavior of the mesh and foam structures produced by this technique, in particular the room temperature tensile and compressive strength and plastic deformation, and compare these results with open cell foam models.
- Extend the technique to the production of other, more complex lattice geometries.

While the first two objectives have been met, meshes and three dimensional foams were not produced due to technical difficulties and delays with the development of the joining system. This work will continue as an unfunded project in the PI's research group.

Status of Effort

The electromechanical forming system was completed in FY 2009. Initial assessment of the equipment capabilities indicates that it will provide added value to the joining experiments that was not available with existing thermomechanical test frames (e.g. Gleeble). Work which will fully utilize this equipment, both to construct the proposed mesh and foam structures and as an alternative frame for thermomechanical testing, will continue under different funding sources.

During the design and construction of the custom equipment, we have used alternative means to characterize the electromechanical joining process on simple BMG joint geometries. We have continued our analysis of the nature of bonding at joint interfaces, as introduced in our 2007 report. We have shown that there is metallurgical bonding at the interface even when interpenetration is limited, although the cohesion is incomplete across the entire interface. Interpenetration and mechanical interlocking does not significantly increase the level of cohesion or the bond strength. The degree of joining appears to increase as the stress during joining decreases. Further analysis suggests that the local strength of the joined sections of the interface approaches ½ the bulk strength of the alloy. Recent efforts have focused on varying the stress state at the interface in order to evaluate the effect of shear on local plastic flow and interface cohesion. Initial experiments have been inconclusive, and further testing is in progress.

Finally, in collaboration with researchers at Cambridge University, we have evaluated a gallium surface treatment for its effectiveness on breaking up the surface oxide layer and promoting cohesion at the interface during diffusion bonding and the proposed electromechanical joining process. The gallium surface treatments have shown promise in the successful diffusion bonding of aluminum alloys and stainless steel alloys [1]. However, in the present study the gallium had a negative effect on the joining process. Treated surfaces did not join, even under processing conditions which produced successful joints with untreated specimens.

Technical Approach and Accomplishments

Introduction

Bulk metallic glasses (BMGs) represent a revolutionary new class of engineering materials with the potential to vastly improve the performance and reliability of Air Force systems [2, 3]. These fully metallic materials exhibit extraordinary tensile strengths, large elastic deflections, and fracture toughness values an order of magnitude higher than traditional glasses. Due to their unique, disordered atomic structure, BMGs soften considerably at elevated temperatures prior to melting. This "homogeneous flow" at low stresses permits the use of inexpensive polymer molding and forming techniques, previously unheard of for high strength materials. Such inexpensive manufacturing techniques give BMGs an additional competitive advantage over traditional alloys.

High volume utilization of BMG components, particularly in safety critical applications, requires the capability for generalized plastic flow at room temperature. At low temperatures, flow in BMGs is highly localized in shear bands. Techniques to distribute flow in multiple shear bands are of significant interest. Recent observations have revealed a length scale dependence for shear band formation, which may hold the key to improving room temperature plasticity. The strain to failure increased by two orders of magnitude as the thickness of the structure decreased from 1 mm to 80 μm due to a dramatic increase in shear band density [4]. This suggests that foams, with cell wall or strut thicknesses of ~100 μm, may exhibit a high shear band density and large scale plasticity at room temperature. Indeed, up to 50% plastic strain has been observed in compression for a Zr-based BMG foam with relative density of 28% [5]. Combined with the already high strength of the alloys, it may be expected that BMG foams will exhibit extraordinary energy absorption capabilities, making them suitable for light weight armor applications. Furthermore, it is well known that metallic foam sandwich structures offer significant increases in strength and stiffness while saving weight relative to solid structures, making them ideal for a variety of aerospace structural applications. Due to the impressive mechanical properties of the fully dense material, BMG foams have the potential to outperform other metallic foam systems.

The present research program focuses on developing a solid state joining technique to create open cell BMG foams with excellent microstructural control. These "periodic cellular materials" (PCMs) consist of periodic arrays of struts or prismatic members, each oriented for optimal load transfer without failure, in contrast to stochastic (random) foams. Crystalline metallic PCMs have been produced by investment casting techniques and by the welding of pre-fabricated wire meshes, both of which involve working in the liquid state and may be inappropriate for maintaining the glassy structure. The current work instead takes advantage of the excellent formability of BMGs in the super-cooled liquid state to join the struts.

In the present work, BMG joining is accomplished by an electromechanical technique. Previous work has shown that, due to the relatively high electrical resistivity of glassy alloys, an applied current can heat the glass to its supercooled liquid regime (Joule heating) without heating the surrounding molds or dies. The addition of a small compressive stress to stacked and heated BMG beams results in homogeneous flow at the point of contact and ultimately interpenetration of the beams [6]. This research program sought to identify the processing parameters required to produce successful joints, including the magnitude of the applied stress, interface surface roughness, and the relative importance of shear vs. normal loading.

Summary of Previous Work

FY 2007 and 2008 work focused the design of a custom electromechanical joining system (described below) and creating model joints using existing electro-thermo-mechanical test frames (Gleeble and Instron ETMT) which operate according to the same principle as the proposed Joule heating technique. We observed that it was possible to create a cohesive joint in a non-interpenetrating specimen geometry. However, while the glass clearly softened and significant plastic deformation occurred on either side of the joint interface, the joining was incomplete, with a significant fraction of the interface area unaffected. We observed that the joined area as well as the nominal joint tensile strength increased as the joining pressure decreased. This decrease in the effectiveness of the joining process with increasing pressure was attributed to poorer interfacial contact at the lower pressure, increasing the electrical resistance and therefore the temperature at the interface. Furthermore, we hypothesized that shear strains play an important role in the joining process. As the interface was compressed, it expanded significantly in the lateral directions. At higher joining stresses (i.e. stress normal to the interface), friction across the interface prevented the faces from sliding past each other (i.e. the faces "stick" due to friction), whereas at lower joining stresses, there is less friction and more "slipping" can occur. This dynamic slipping process may result in more energy being deposited at the interface, increasing deformation locally, breaking up the surface oxide, and enhancing joining. The role of shear stresses at the interface was investigated in FY 2009, as described below.

The model joining work extended to interpenetrating joints. We observed that high strain rates impeded joint formation – the only successful joints were produced at lower strain rates. Interpenetration significantly enhanced the strength and deformation characteristics of the joint. Whereas the non-interpenetrating joints were essentially linear elastic to failure when tested in tension, the interpenetrated joints exhibited peak strengths of approximately 1 GPa (~½ the bulk yield strength) and significant energy dissipation associated with work against mechanical interlocking. The details of the failure process varied greatly, however, with some failure surfaces being very rough and others relatively smooth. The details of the joining and failure mechanisms are as yet unclear, and were the subject of further experiments conducted in FY 2009.

FY 2009 Progress

Completion of the Custom Electromechanical Joining System

The development of an integrated joining apparatus was, in part, to eliminate some of the previous limitations in the experimental setup posed by both the Gleeble and the Instron ETMT. The joining equipment was designed to utilize the high current capability of the Gleeble system with the testing flexibility and sensitivity of the Instron unit. In addition to maximizing the benefits of each system, the joining equipment has a larger vacuum chamber and the MTS TestWare software utilized on the MTS 810 test frame is more robust than what is available on the Instron unit.

The custom electrical (heating) system for the joining equipment was necessary due to the requirement of large current densities to produce joined structures as described in the project proposal. Previous work [5] showed that successful joining of BMG components could occur at current densities on the order of 130 A/mm^2. The project objectives include the development of a BMG mesh by an electromechanical joining technique. To produce a 50% dense 2 cm^2 mesh would require approximately 13,000 A. The development of the custom joining equipment was based on the 13,000 A requirement. The following major components necessary for the construction of the custom electromechanical joining system were received (Figure 1) and assembled:

- 14 in diameter, single walled environmental chamber (Oxygon). The chamber is capable of high vacuum operation, but it is anticipated that all joining will be conducted either under a low vacuum backfilled with argon or in air.
- Welch 1402, 5.6 CFM vacuum pump.
- 55 KVA, 3.2 V to 5.2 V power supply (Taylor Winfield) with a rectifier assembly and a Unitrol Solution (9180M-800) controller with a 300 amp silicon controlled rectifier (SCR) and constant current monitoring.
- Refrigerated heat exchanger (Opti Temp) to provide cooling for the electrical equipment and the joining platens inside the vacuum chamber.

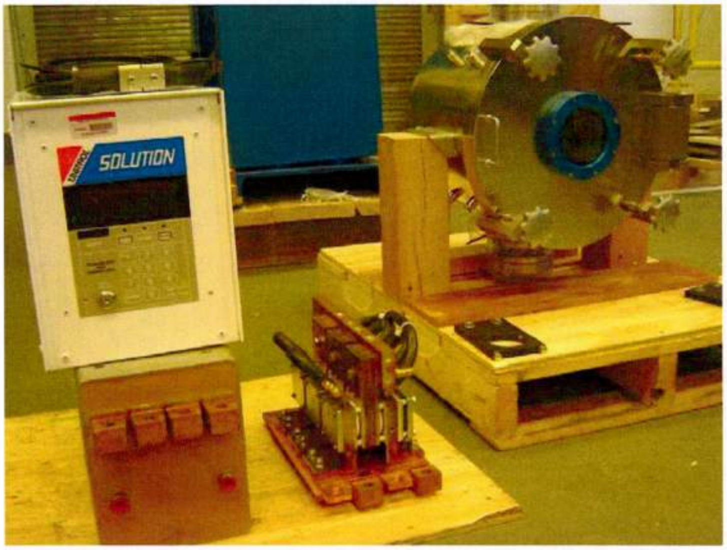
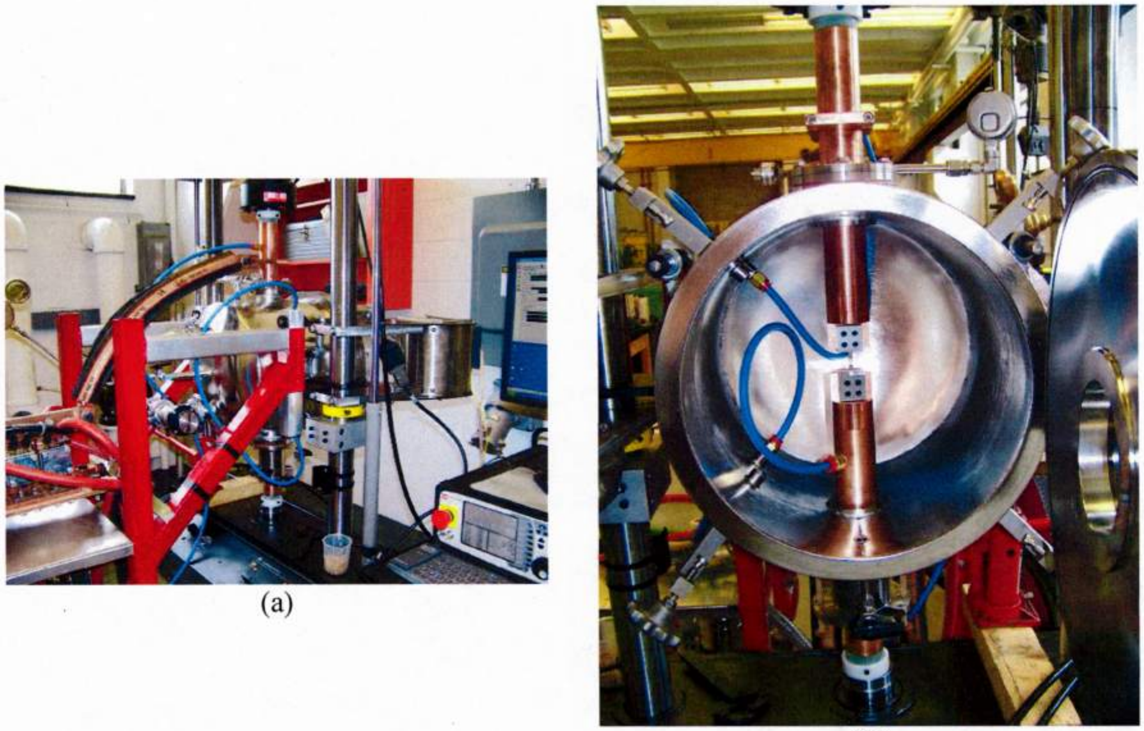


Figure 1. Controller, power supply, and environmental chamber for electromechanical joining system.

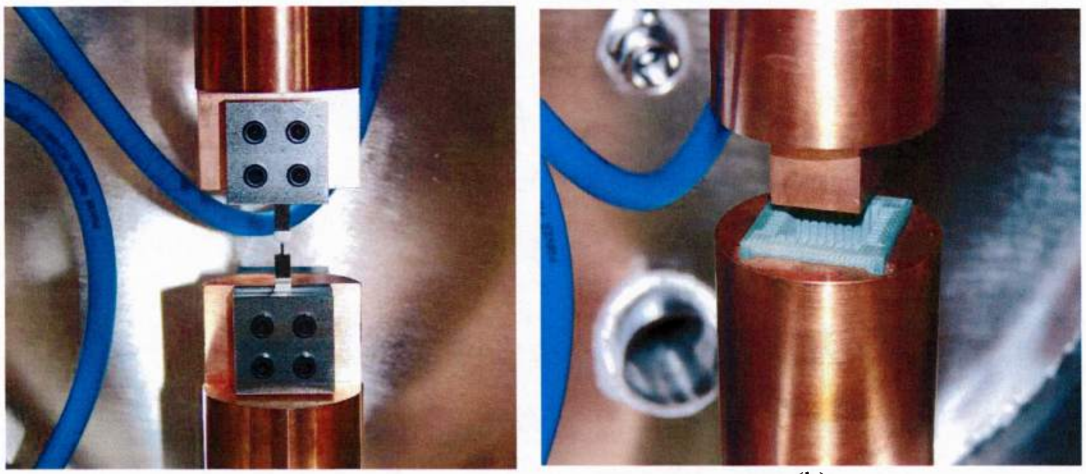
The joining equipment was specifically designed to attach to and work in conjunction with an MTS 810 mechanical test frame (Figure 2). The MTS mechanical test frame allowed for a wide range of mechanical testing conditions (tension and compression) and the acquisition of data from external sources, specifically temperature. The joining equipment is capable of applying current and Joule heating material in three different control modes: constant current, constant voltage, and constant temperature as read by an infrared thermometer. The inclusion of the IR thermometer should eliminate the difficulties associated with using traditional thermocouples for monitoring and controlling the joining process in the constant temperature mode. Two sets of electrode dies have been produced (Figure 3). A vise grip set is designed to hold modified dogbone specimens similar to those used in the Gleeble and ETMT studies. Note that these grips are designed for use in either compression or tension and could therefore be used for tension testing previously joined or monolithic specimens at elevated temperatures. A second die is designed to hold small beams or wires in position for the production of meshes.



(a)

(b)

Figure 2. Joining equipment attached to an MTS 810 mechanical test frame. The joining chamber and rectifier set as seen from the front of the test frame (a) and the chamber interior as seen from the rear of the test frame (b).



(a) (b)

Figure 3. Electrode/die sets designed and fabricated for various test conditions: a single specimen, "vise" type configuration (a) and set for producing flat mesh geometries (b).

Operating programs for the power supply controller have been developed and debugged. With the IR thermometer installed, several tests were conducted with monolithic specimens to identify the proper parameters to achieve a quick ramp to a hold at a preset temperature. The debug tests show that the equipment exceeds the heating capacities of the Gleeble and ETMT systems previously used for the joining experiments. As shown in Figure 4, the debugging process resulted in some specimens rapidly heated in excess of the melt temperature (~ 827°C, 13a and 13b), with later specimens remaining intact as the target temperature was achieved.

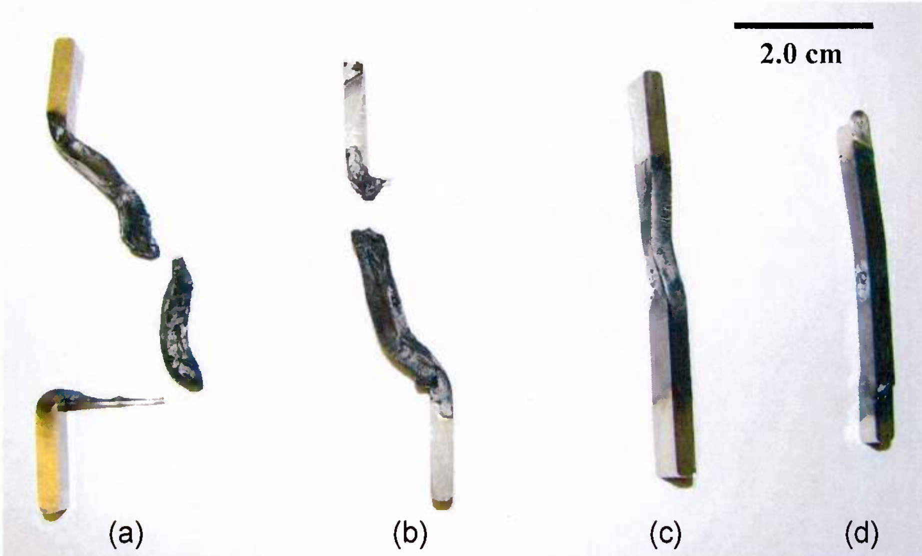
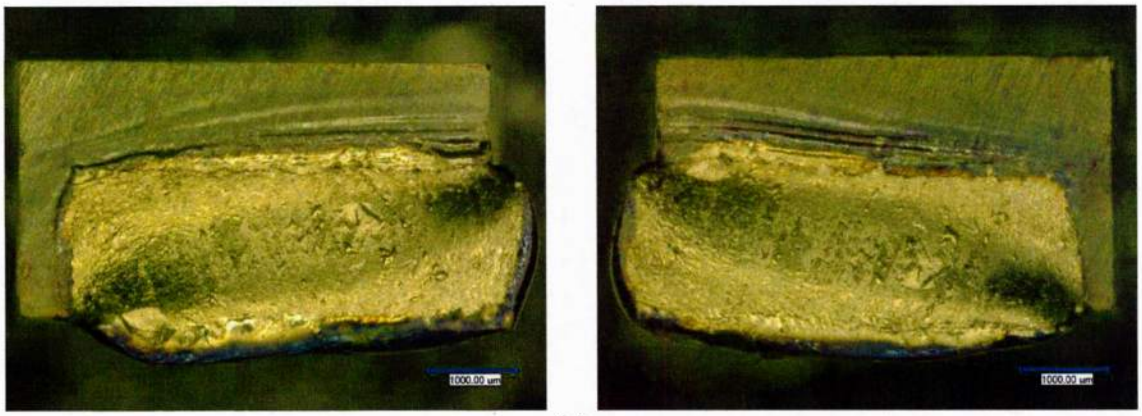


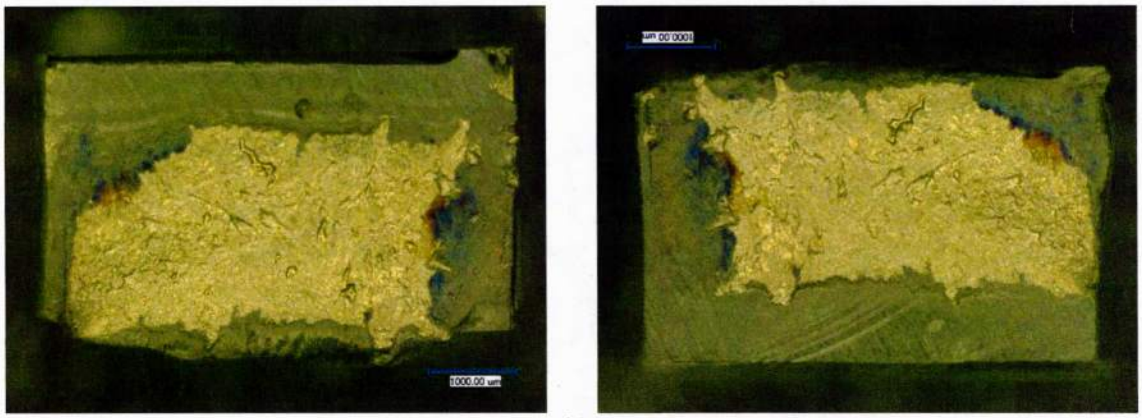
Figure 4. Metallic glass beams used to debug the new joining equipment in constant temperature mode. The first two specimens (a) and (b) were heated in excess of 827°C in less than 2 seconds, resulting in rupture. The final two trials were ramped to 500°C in 2 seconds and maintain ± 50°C for an additional 4 seconds.

Following the debugging process, two attempts were made to join non-interpenetrating specimens with 3mm x 5mm cross sections. In both cases, the specimens were preloaded to 1 kN (~67 MPa assuming perfect alignment) in compression, and the load was allowed to relax as the material was heated to 482°C (900°F), as indicated by the IR thermometer. Each specimen joined, but broke apart as the specimens were removed from the grips due to an unexpected twisting of the specimen as the grips were loosened (Figure 5). New grips which will allow the specimen to be removed without the potential application of torque have been designed and will be implemented.

The new joining equipment was designed to provide flexible and robust experimental opportunities. Table 1 below summarizes the capabilities of the new system and compares them with those of the Gleeble and Instron ETMT systems used in prior testing. The table also summarizes the current state of verification of each of the capabilities.



(a)



(b)

Figure 5. Two specimen joined during the equipment setup and debugging process. Each of the joined specimens broke at the interface during removal from the equipment.

Table 1. Summary of proposed and actual joining equipment as compared to experimental capabilities of the Gleeble and Instron ETMT systems.

	Proposed capabilities	**Actual capabilities**	**Gleeble**	**Instron (ETMT)**
Tension	✓	✓	✓	✓
Compression	✓	✓	✓	✓
Torsion			✓	
Constant Temperature	✓	✓	✓	✓
Constant Current	✓	untested		✓
Constant Voltage	✓	untested		
Flexible programming	MTS Testware	MTS Testware		
Current limitation	> 13,000 A	> 13,000 A	> 13,000 A	60A
Sample size limitations	31 cm diameter test chamber	~ 20 cm work area	~ 10 cm work area	3 cm work area

Stress State Effects on Electromechanical Joining

Experimental

In order to investigate the effect of combined shear and normal loading on the interface during joining, we attempted to join specimens with angled interfaces. Specimens were prepared from 3 mm diameter cast rods of $Zr_{58.5}Nb_{2.8}Cu_{15.6}Ni_{12.8}Al_{10.3}$ (nominal at. %) bulk metallic glass. The 3 mm diameter rods were sectioned into pairs with interface orientations at 45° and 90° to the tensile axis. A flat reference surface was ground into the sides of the 45° specimens to assist with alignment. Six sets of specimens were prepared at each orientation. To remove surface damage and prepare the surfaces for joining, the mating surfaces were ground with 400 grit SiC paper. In an attempt to remove or disrupt the surface oxide layer at the joint interface, three specimen sets from both the 90° and 45° sets were prepared using a proprietary gallium surface treatment method provided by collaborators at Cambridge University.

Because the custom system was still in the debugging phase, the joining experiment was conducted using an Instron ETMT. The specimens were clamped to copper grips which also acted as electrodes for the application of current through the specimen. The current resulted in resistive Joule heating of the specimen, primarily at the joint interface where the contact resistance was greatest. The experiments were conducted in an argon environment to minimize oxidation during heating.

For each joining experiment, the specimen halves were brought into contact and the interface was preloaded in compression to 50 N. The pre-loaded specimens were then ramped to the testing load of 200 N in compression, or an axial compressive stress of approximately 28 MPa. The specimens were then heated from room temperature to 725 K (approximately 50 K above the glass transition) at 100 K/s in load control. The load and temperature were held constant for 45 seconds then the current was turned off and the load was reduced to 50 N in compression. The samples were allowed to cool to room temperature under load for an additional 60 seconds prior to removal from the test frame.

Results/Discussion

Conventional Surface Preparation

Of the six sets of specimens prepared without the gallium treatment, none of the specimen with 45° interfaces joined and 2 of the 3 90° interfaces joined. This suggests that shear stresses have a deleterious effect on the joining process – a surprising result since plastic deformation in metallic glasses is thought to be a shear driven process, and the joining mechanism is presumed to be related to material flow. However, it should be noted that the 45° specimens deflected significantly while under load due to the force component on the interface surface perpendicular to the specimen axis. Because the deflection increased with increasing axial load, the axial stress was limited to a value well below that used in prior experiments. Another series of experiments is planned which will better constrain the specimen and permit a wider range of contact stresses. Smaller deviations from 90° will also be examined.

During heating, each of the 6 specimen sets softened near the interface and underwent deformation totaling 2-5.5 mm of total displacement (Figure 6). The two joined specimens (4_90° and 5_90°) underwent appreciably different amounts of deformation. Specimen 6_90° exhibited deformation behavior very similar to 4_90°, but did not join. The reason for this inconsistency is as yet unknown, but it does suggest that the development of cohesion across the interface does not depend directly on the amount of deformation around the interface. It should be noted that the thermocouple on the 6_90° specimen became enveloped by the interface ~50 seconds into the test cycle, which prematurely stopped the heating. We assume that the specimen would have undergone more deformation if the heating cycle had not been interrupted. However, it is unclear if this would have resulted in joint formation

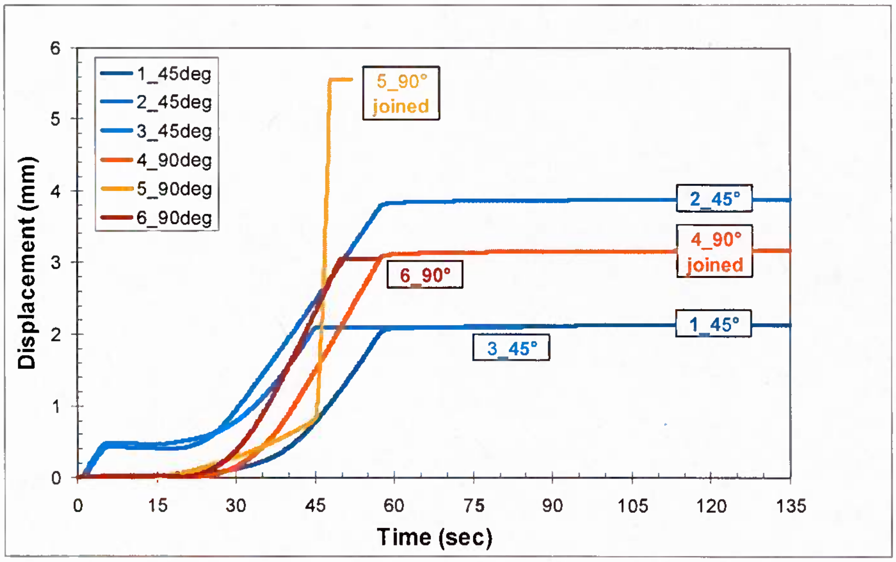


Figure 6. Joining time versus displacement data for surface ground specimens with interfaces oriented 45° and 90° to the loading axis.

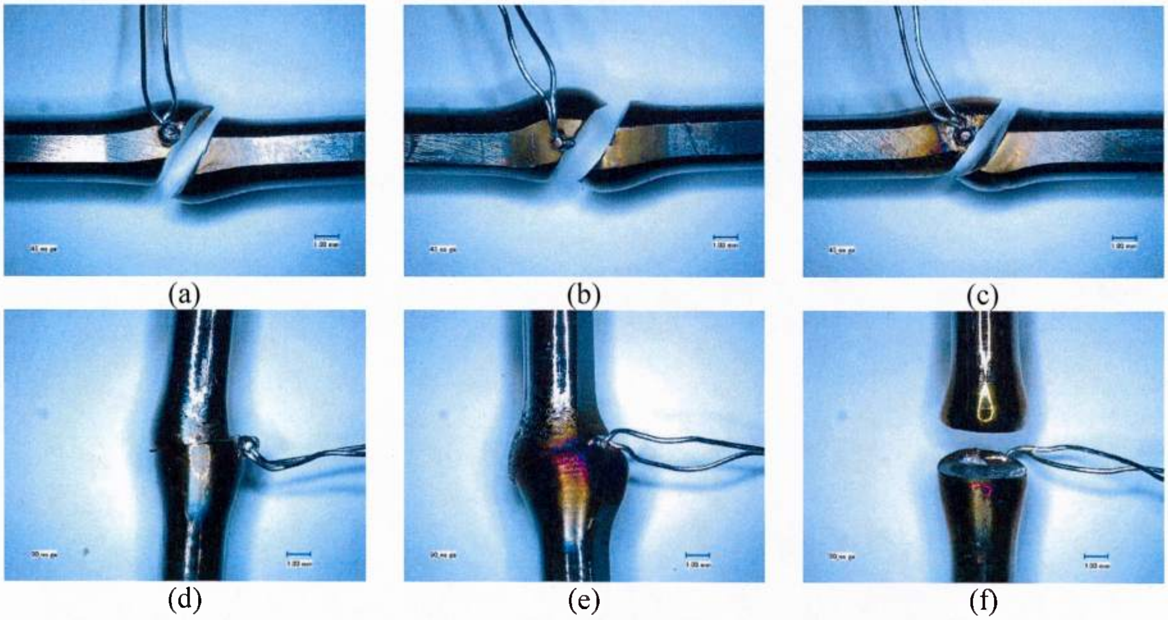
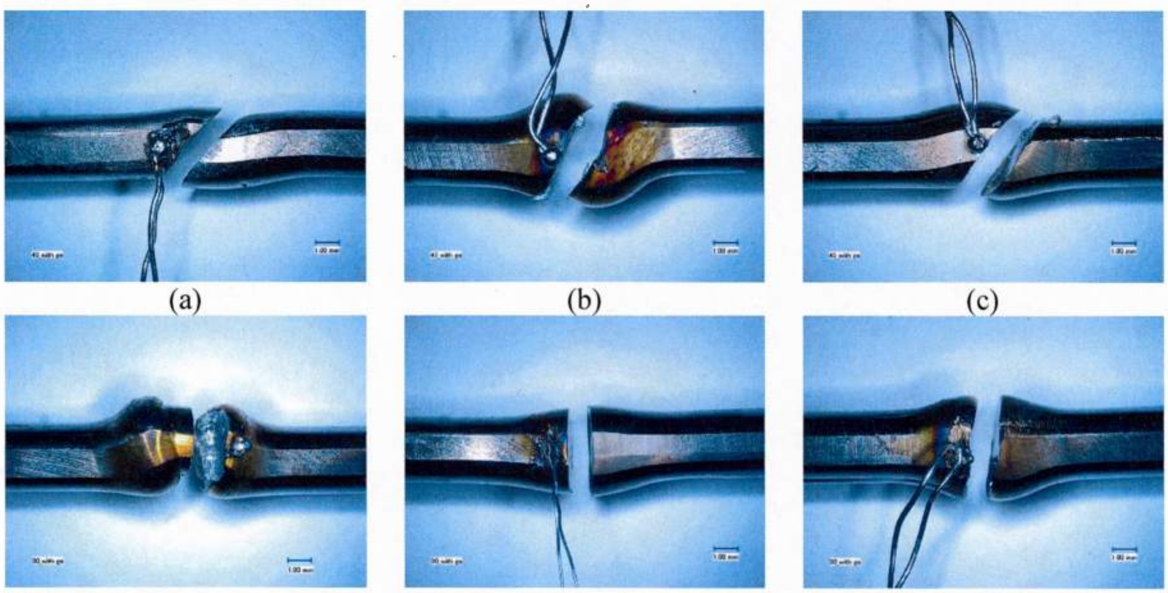


Figure 7. Surface ground specimen prepared with the joint interface orientated 45° (a-c) and 90° (d-f) with respect to the loading axis.

Gallium Surface Preparation

Prior work by Shirzadi and coworkers at Cambridge University has suggested that a gallium surface treatment is effective at improving the quality of diffusion joints in materials that produce stable surface oxide films, such as aluminum and superalloys [7-9]. It is believed that this surface treatment removes the surface oxide, permitting intimate contact between the surfaces to be joined. The Zr-based glass used in this study also forms a thin, very stable surface oxide which may impede bonding. However, in the present study neither the 45° or 90° specimens prepared with the gallium surface treatment joined (Figure 8). SEM analysis of the specimen surfaces is ongoing. We believe that excess gallium may have inhibited wetting across the interface or may have diffused into the glass, locally changing its structure and flow behavior.



(a) (b) (c)

(d) (e) (f)

Figure 8. Both the 45° (a-c) and 90° (d-f) specimens prepared using a gallium surface treatment did not join.

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9. Shirzadi, A.A. and E.R. Wallach, *Novel method for diffusion bonding superalloys and aluminium alloys (USA patent 6,669,534 B2, European patent pending).* New Frontiers of Processing and Engineering in Advanced Materials, 2005. 502: p. 431-435.

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Nick Hutchinson	Graduate Research Assistant, OSU
Chris Eastman	Undergraduate Research Assistant, OSU

Publications

None (3 in preparation)

Interactions/Transitions

<u>Conference Presentations</u>

1. N. Hutchinson, J. Bennett, and K.M. Flores, “Solid-State Joining of Based Bulk Metallic Glass”, 2007 Fall Meeting of the Materials Research Society, Boston MA, November 2007.
2. N. Hutchinson, Y. Zhang, J. Bennett, G. S. Daehn and K. M. Flores, “Solid-State Joining of a Zr-Based Bulk Metallic Glass”, 2008 TMS Annual Meeting, New Orleans LA, March 2007.

3. N. Hutchinson and K. M. Flores, "Solid State Joining of Bulk Metallic Glass via Mechanically Assisted Diffusion", 2008 Fall Meeting of the Materials Research Society.
4. N. Hutchinson, Y. Zhang, G. S. Daehn and K. M. Flores, "Characterization of Local Deformation during Low and High Strain Rate Joining of Bulk Metallic Glasses", 2009 TMS Annual Meeting.
5. N. Hutchinson, K. M. Flores, "Effect of Stress State on Flow of Bulk Metallic Glass Interfaces", 2010 TMS Annual Meeting, abstract submitted 2009.

New Discoveries, Inventions, or Patent Disclosures

None

Honors/Awards

None